Crystal structure of ($\pm$)-1-adamantyl (1*R*,2*S*)-2-hydroxy-2-phenyl-3-cyclopentene-1-carboxylate, C₂₂H₂₆O₃

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Abstract

C₂₂H₂₆O₃, triclinic, $P\bar{1}$ (No. 2), $a = 9.834(2)$ Å, $b = 10.629(2)$ Å, $c = 9.754(5)$ Å, $\alpha = 102.21(2)^\circ$, $\beta = 105.49(3)^\circ$, $\gamma = 71.58(2)^\circ$, $V = 923.1$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.044$, $wR_{\text{ref}}(F^2) = 0.148$, $T = 173$ K.

Source of material

To a solution of LiHMDS (0.22 mmol) in diethyl ether (2 ml) was added 1-adamantyl 2-(2-oxo-2-phenylethyl)cyclopropane-1-carboxylate [1,2] (50 mg, 0.15 mmol) in diethyl ether (2 ml) according to the general experimental procedure outlined in [3]. The compound was isolated from hexane solution as colourless rods (yield: 43 mg, 85%; mp = 415 K – 416 K).

Discussion

The structure determination establishes the stereochemistry of the molecule, in particular the relationship between the ester and hydroxyl groups. The near co-planarity of the hydroxyl and phenyl groups at C2, as seen in the O2–C2–C21–C22 torsion angle of $11.4(3)^\circ$, is stabilised by an intramolecular C–H...O interaction so that $d(\text{H}\cdots\text{O2})$ and $d(\text{C22}\cdots\text{O2})$ are 2.33 Å and 2.687(3) Å, respec-

tively. Centrosymmetrically related pairs of molecules associate via O–H...O interactions so that $d(\text{O2}\cdots\text{O11a}^i) = 1.70$ Å, $d(\text{O2}\cdots\text{O11a}^i) = 2.762(3)$ Å and the angle at H = 168° ; symmetry operation $i: 1-x, -y, 2-z$. This association leads to the formation of 12-membered rings.

Table 1. Data collection and handling.

Crystal:	colourless block, size 0.13 × 0.19 × 0.29 mm
Wavelength:	Mo K α radiation (0.71069 Å)
μ :	0.79 cm ⁻¹
Diffraction, scan mode:	Rigaku AFC7R, $\omega/2\theta$
$2\theta_{\text{max}}$:	55°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	4497, 4250
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2\sigma(I_{\text{obs}})$, 1840
$N(\text{param})_{\text{refined}}$:	227
Programs:	teXsan [4], SIR92 [5], SHELXL-97 [6], ORTEPII [7], PLATON [8]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(2)	2i	0.3307	0.0736	0.9416	0.062(2)
H(1)	2i	0.4732	−0.0416	0.6057	0.050(1)
H(3)	2i	0.1217	−0.0104	0.6840	0.050
H(4)	2i	0.2731	−0.2320	0.7012	0.050
H(5A)	2i	0.5212	−0.2415	0.6780	0.062
H(5B)	2i	0.5180	−0.1778	0.8430	0.062
H(22)	2i	0.2665	0.3342	0.7654	0.050
H(23)	2i	0.1811	0.4832	0.5980	0.050
H(24)	2i	0.1139	0.4083	0.3542	0.050
H(25)	2i	0.1341	0.1817	0.2778	0.050
H(26)	2i	0.2206	0.0319	0.4443	0.050
H(41A)	2i	0.5557	0.3502	0.6685	0.062
H(41B)	2i	0.6959	0.2516	0.6103	0.062
H(42)	2i	0.7303	0.4706	0.6873	0.050
H(43A)	2i	0.9447	0.2862	0.7155	0.062
H(43B)	2i	0.9662	0.4066	0.8422	0.062
H(44)	2i	1.0422	0.2035	0.9398	0.050
H(45A)	2i	0.8854	0.0875	0.7640	0.062
H(45B)	2i	0.8674	0.0838	0.9219	0.062
H(46A)	2i	0.6493	0.2395	1.0137	0.062
H(46B)	2i	0.5269	0.3427	0.9148	0.062
H(47)	2i	0.6835	0.4585	1.0908	0.050
H(48A)	2i	0.5871	0.5418	0.8658	0.062
H(48B)	2i	0.7470	0.5637	0.9337	0.062
H(49A)	2i	0.8971	0.2738	1.1181	0.062
H(49B)	2i	0.9369	0.3995	1.0886	0.062

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Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
O(2)	2i	0.3196(2)	0.1399(2)	0.8678(2)	0.049(1)	0.0396(9)	0.0315(9)	-0.0137(8)	0.0126(7)	-0.0013(7)
O(11)	2i	0.5984(2)	0.1235(2)	0.7346(2)	0.0340(8)	0.0353(8)	0.0345(9)	-0.0147(7)	0.0071(7)	0.0044(7)
O(11A)	2i	0.6589(2)	0.0013(2)	0.9163(2)	0.044(1)	0.049(1)	0.041(1)	-0.0175(8)	-0.0011(8)	0.0153(8)
C(1)	2i	0.4675(2)	-0.0309(2)	0.7085(2)	0.035(1)	0.031(1)	0.034(1)	-0.010(1)	0.008(1)	0.003(1)
C(2)	2i	0.3128(2)	0.0664(2)	0.7267(2)	0.034(1)	0.034(1)	0.034(1)	-0.011(1)	0.011(1)	0.001(1)
C(3)	2i	0.2258(3)	-0.0338(3)	0.7032(3)	0.037(1)	0.051(2)	0.046(2)	-0.021(1)	0.012(1)	0.002(1)
C(4)	2i	0.3100(3)	-0.1571(3)	0.7122(3)	0.053(2)	0.044(2)	0.042(2)	-0.027(1)	0.010(1)	0.001(1)
C(5)	2i	0.4700(3)	-0.1664(2)	0.7417(3)	0.052(2)	0.033(1)	0.047(2)	-0.015(1)	0.009(1)	0.006(1)
C(11)	2i	0.5868(2)	0.0297(2)	0.7999(3)	0.032(1)	0.030(1)	0.038(1)	-0.008(1)	0.011(1)	0.002(1)
C(21)	2i	0.2536(2)	0.1672(2)	0.6218(2)	0.025(1)	0.038(1)	0.039(1)	-0.005(1)	0.009(1)	0.004(1)
C(22)	2i	0.2403(3)	0.3022(2)	0.6661(3)	0.037(1)	0.044(2)	0.051(2)	-0.015(1)	0.005(1)	0.008(1)
C(23)	2i	0.1890(3)	0.3908(3)	0.5665(3)	0.045(2)	0.044(2)	0.079(2)	-0.011(1)	0.008(2)	0.023(2)
C(24)	2i	0.1492(3)	0.3468(3)	0.4220(3)	0.045(2)	0.068(2)	0.068(2)	-0.000(2)	0.011(2)	0.038(2)
C(25)	2i	0.1613(3)	0.2131(3)	0.3771(3)	0.051(2)	0.069(2)	0.042(2)	0.004(2)	0.008(1)	0.016(2)
C(26)	2i	0.2129(3)	0.1242(3)	0.4764(3)	0.046(2)	0.046(2)	0.038(1)	0.001(1)	0.010(1)	0.005(1)
C(40)	2i	0.6878(2)	0.2155(2)	0.8086(2)	0.030(1)	0.035(1)	0.042(1)	-0.014(1)	0.010(1)	0.001(1)
C(41)	2i	0.6619(3)	0.3056(3)	0.6969(3)	0.054(2)	0.048(2)	0.048(2)	-0.023(1)	0.018(1)	0.005(1)
C(42)	2i	0.7465(3)	0.4113(3)	0.7607(3)	0.067(2)	0.055(2)	0.069(2)	-0.035(2)	0.020(2)	0.010(2)
C(43)	2i	0.9098(3)	0.3398(3)	0.8019(4)	0.064(2)	0.073(2)	0.098(3)	-0.047(2)	0.040(2)	-0.017(2)
C(44)	2i	0.9349(3)	0.2490(3)	0.9122(4)	0.031(1)	0.057(2)	0.093(2)	-0.019(1)	0.009(2)	-0.001(2)
C(45)	2i	0.8508(2)	0.1431(2)	0.8497(3)	0.030(1)	0.044(2)	0.064(2)	-0.011(1)	0.014(1)	-0.005(1)
C(46)	2i	0.6333(3)	0.2983(2)	0.9413(3)	0.035(1)	0.038(1)	0.039(1)	-0.011(1)	0.011(1)	0.001(1)
C(47)	2i	0.7182(3)	0.4037(2)	1.0035(3)	0.051(2)	0.041(1)	0.052(2)	-0.017(1)	0.012(1)	-0.005(1)
C(48)	2i	0.6929(3)	0.4953(3)	0.8927(3)	0.061(2)	0.039(2)	0.074(2)	-0.023(1)	0.015(2)	0.002(1)
C(49)	2i	0.8809(3)	0.3327(3)	1.0457(3)	0.050(2)	0.057(2)	0.067(2)	-0.030(1)	-0.006(1)	-0.002(2)

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